

## Article

# Soil Characteristics and Fertility of the Unique Jarrah Forest of Southwestern Australia, with Particular Consideration of Plant Nutrition and Land Rehabilitation

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**Abstract:** The jarrah forest is a natural ecosystem with significant endemism in the flora and fauna. The forest stands on the western edge of the ancient Great Plateau of Western Australia on the granitic shield of the Yilgarn craton (2.5 Gya). The long-term migration of soluble iron and aluminium led to the formation of bauxite ore. The regolith ore is bound by surficial topsoil and deep pallid zone kaolinite clays, primarily used in land rehabilitation. We investigated the chemical fertility of the substrates, along with key physical properties. We found the capacity of these soils to provide a stable growth medium differed considerably in their ability to retain and supply nutrients. These substrates are deficient in nitrogen, phosphorus, and micronutrients. In the topsoil, 15% of total P is plant-available, while in the pallid zone layer, only 1% of total P is available. <sup>31</sup>P-NMR showed no organic P in the pallid zone, whereas the topsoil had significant organic P and, consequently, a supply of phosphate ions. This shows the importance of organic P in the topsoil for ecosystem nutrition when inorganic fertilisers are not applied in state-of-the-art restoration schemes.

**Keywords:** Archean; bauxite; aluminium ores; kaolinite; NMR spectroscopy; micronutrients; boron; copper; zinc



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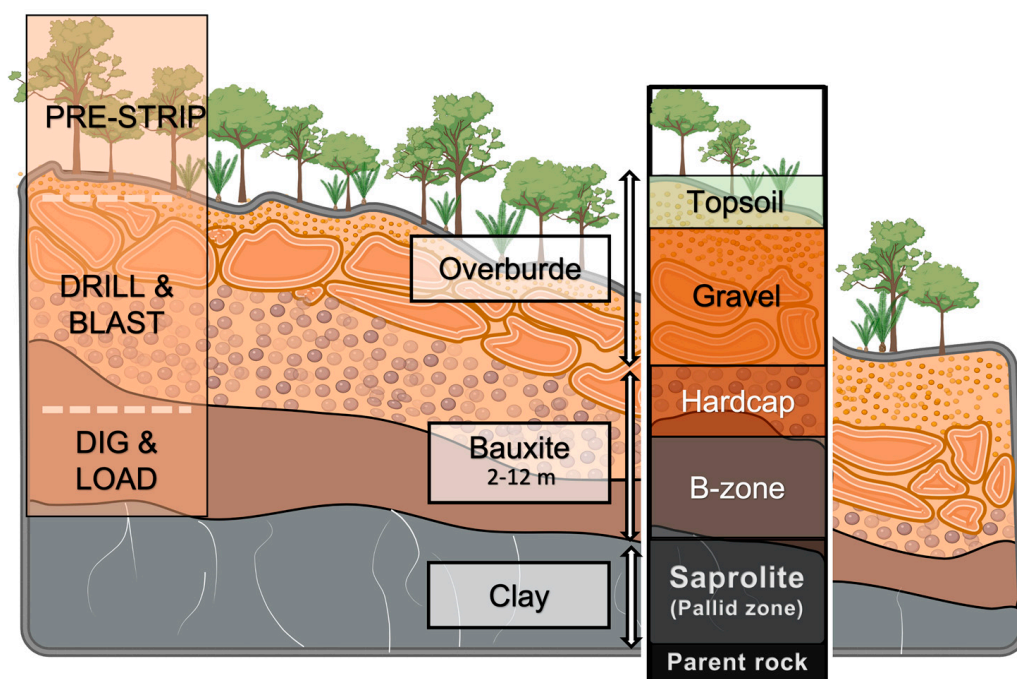


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## 1. Introduction

The jarrah forest in Australia is a natural ecosystem with significant endemism in the flora and fauna [1]. It is within one of the world's biodiversity hotspots [2], where the soils are deeply weathered and consequently P-impooverished [3]. Many of the hundreds of understory species found in the forest have specialized adaptations for P-acquisition, including cluster roots, mycorrhizal symbiosis, and the exudation of carboxylases [4]. The dominant overstorey species are *Eucalyptus marginata* (jarrah) and *Corymbia calophylla* (marri). The mid-storey layer is dominated by *Banksia grandis*, *Allocasuarina fraseriana*, *Xanthorrhoea preisii*, *Bossiaea aquifolium*, and various *Acacia* species, with a diverse understory. The forest stands on the western edge of the Great Plateau of Western Australia [5]. The plateau is based on a mainly granitic shield of the Archean age (2.5 billion years) of the Yilgarn craton, which has remained tectonically stable over a long period of geological time. The evolution of regolith in the Darling Range has been influenced mainly by two climatic regimes: first, seasonally humid, subtropical to tropical conditions during the Cretaceous to Middle Miocene and, second, drier climates since the Miocene that have continued to the present [6]. The former resulted in extensive deep lateritic weathering with high water tables in acidic environments and the latter caused a lowering of water tables, slowing chemical weathering and erosional modifications of the land surface in alkaline conditions. During the recent arid phase, gypsification, silicification, and carbonate precipitation have

been the main pedogenic processes [7]. Soils in the Darling Range are generally shallow in the upslope areas with lateritic duricrust and granitic bedrock at or close to the surface. The mid-slope regions are dominated by shallow soil with lateritic duricrust close to the surface, and sandy piezolithic gravel dominates the upper strata. Clayey sandy silt has been deposited by alluvial processes in the low-lying areas (Figure 1). The resultant soils are typical of natural habitats in old, climatically buffered, infertile landscapes [3], with scarce plant-available nutrients reflected in low foliar tissue concentrations [8].



**Figure 1.** Darling Range bauxite profile.

After 2.5 billion years of the migration of soluble iron (Fe) and aluminium (Al) released from the weathering of secondary clay minerals in the upper soil horizons, as well as their accumulation in subsoil horizons, jarrah forest bauxite ore bodies were formed [9]. These ore bodies of 1 to 100 ha are defined mainly on the basis of available Al and reactive silica content. Based on world standards for bauxite ores, Al content is low, averaging about 30%; however, they are still economical to process given their low reactive silica contents (1–2%) [10].

Australia is the world's sixth largest aluminium producer, producing 1.56 million tonnes in 2021, of which 1.43 million tonnes was exported. Bauxite mining plays an important role in Australia's economy, worth over AUD 3 billion in export earnings in 2021, according to the Australian Bureau of Statistics. Australia has decided to sacrifice some of its unique natural ecosystems in order to maintain economic prosperity for its people. Bauxite mining, a form of strip-mining, can result in the complete destruction of the existing ecosystem, and the scale of land disturbance is large (ca. 500 ha per year per mine) compared with that of most other mining types. In order to minimise the negative effect of bauxite mining, the state and federal governments have legislated strict measures, ensuring that mining companies restore natural forests into sustainable ecosystems that reflect the original forest prior to mining. It seems that these strategies have been successful in terms of the recovery of many, but not all, e.g., [4,11,12], ecosystem properties based on various ways of measuring the success of restoration [13].

In the rehabilitation process, immediately before mining, the topsoil (A and upper B horizons, ~0.1 to 0.3 m) is removed from mining areas and placed directly on previously mined areas already prepared to receive topsoil before seeding [14–16].

Deep and intensive weathering in jarrah forest soils has led to the depletion of phosphorus (P). Restoring productivity and replenishing soil nutrients lost during the mining process are generally the main goals of restoration programs, which should be achieved through minimal management or input. Soil fertility in these rehabilitated areas is addressed through fertiliser application in order to initiate the incipient ecosystem [17–20]. There is currently a paucity of information on essential soil properties that allow optimal soil management and fertilisation regimes. There is a fundamental lack of unambiguous chemical fertility and key physical properties for these soils published in the literature. The topsoil and deeper pallid zone kaolinites make up the majority of the potential plant growth medium. Topsoils are used in numerous restoration schemes, e.g., [13,21,22], and pallid zone clays are used extensively in the construction of berms and batters of vegetated tailing storage facilities [23–25]. Based on this, we investigated, in detail, the chemical fertility of topsoil and pallid zone saprolite clay layers supplemented with some key physical properties. This allowed us to consider the capacity of these soils to provide a stable growth medium and retain and supply nutrients to provide a physically stable and chemically fertile landscape for revegetation.

## 2. Materials and Methods

### 2.1. Sampling of Soil Materials

In this study, two jarrah forest soil layers from the Darling Plateau in the southwest of Western Australia were evaluated: a sandy topsoil (0–15 cm) and a saprolitic clay from a depth in the regolith (>12 m). The samples were taken from a bauxite mine near the town of Boddington, 130 km south-east of Perth with a latitude of 33°12' S and longitude of 116°4' E. These we refer to as the topsoil and pallid zone layers, respectively. The region has a Mediterranean climate with a mean annual rainfall of 1200 mm, an average summer monthly maximum temperature of 28 °C, and an average winter minimum of 5 °C. Four representative samples of each soil layer were collected. A wide range of chemical and physical analyses was completed on all samples along with more advanced analysis using solution <sup>31</sup>P nuclear magnetic resonance (NMR) spectroscopy and phosphorus adsorption isotherms. All analyses were replicated three times unless stated otherwise. Where appropriate, soils were air-dried (35 °C) to a constant weight and sieved (<2 mm) prior to analysis.

### 2.2. Chemical Properties of Soils

The pH and electrical conductivity (EC) were measured on air-dried and sieved soils from all samples, and three replicates of each sample were tested. Electrical conductivity and pH measurements were made on a 1:5 soil:water extract, with the pH also measured on a 1:5 soil:CaCl<sub>2</sub> (0.01 M) extract [26]. The soil and solution were tumbled for 1 h before being centrifuged at 3000 rpm for 5 min to remove the suspended material. The pH and EC of the supernatant were tested.

Soil organic carbon was determined in one sample of each soil layer using the Walkley and Black method [27], which is based on the oxidation of organic matter via dichromate in the presence of H<sub>2</sub>SO<sub>4</sub>.

Samples were analysed for their major element concentrations using the X-ray fluorescence (XRF) of fused beads. This involved fusing ground samples at a high temperature with lithium metaborate/tetraborate and forming it into a glass bead. Samples were then analysed on a Philips PW1404 XRF instrument. The instrument was calibrated prior to use with a range of international standard reference materials, and the calibration was re-sloped on a daily basis using a monitor sample. Additionally, an in-house reference material was analysed with the test materials. Whereas the element was analysed as an oxide (Al, Si, Ti, Fe, Mn, Ca, K, Mg, and P), the concentration of the element itself was calculated through basic stoichiometry.

Samples of each soil layer were also analysed using X-ray diffraction (XRD) to determine their mineralogy. A Philips PW1830 X-ray diffractometer with a Cu target tube

was used. Mounts of finely ground material were prepared, and diffraction patterns were determined for the range 2–70° at a scan rate of 0.02. For mineral identification, the spacing and intensities were compared with those for the appropriate minerals given in JCPDS data files.

The ammonium and nitrate ( $\text{NO}_3^-$  and  $\text{NH}_4^+$ ) were analysed in soil samples after extraction with 2 M KCl (1:10 soil-to-solution ratio) and then determined colourimetrically using the method of Keeney and Nelson [28].

The assessments of cation exchange capacity and exchangeable sodium percentage were outsourced to CSBP laboratories. The Gilman and Sumpter method [26] was used, employing a 0.1 M  $\text{BaCl}_2$ /0.1 M  $\text{NH}_4\text{Cl}$  extraction of exchangeable bases. The exchangeable sodium percentage (ESP) was calculated from the effective cation exchange capacity (ECEC). ESP is simply the percentage of exchangeable sodium ions that make up the total ECEC and indicates soil sodicity.

Available P and K were determined using the Colwell bicarbonate extraction method [29]. Labile and adsorbed levels of S were determined using the KCl method [30]. Plant-available levels of Fe, Cu, Zn, and Mn were measured via extraction with DTPA.

### 2.3. Phosphorus Sorption Isotherm

Phosphorus sorption was determined by equilibrating 3 g soil samples with 30 mL of 0.01 M  $\text{CaCl}_2$  containing one of several concentrations of P as  $\text{KH}_2\text{PO}_4$  in 50 mL plastic centrifuge tubes. The initial solution P concentrations varied between 0 and 60  $\mu\text{g mL}^{-1}$  in order to achieve final equilibrium concentrations up to 1.5  $\mu\text{g mL}^{-1}$ . The samples were equilibrated for a 24 h period. A few drops of toluene were added to suppress microbiological activity, and the samples were gently shaken on an end-over-end shaker at 10 rpm. At the end of the equilibration period, the solution was separated using a combination of centrifugation and filtration (Whatman #40). The concentration of P in solution was determined with the molybdate–ascorbic acid method [31]. The amount of P sorbed was calculated as the difference between the amount of P added and remaining in solution [32].

The Langmuir equation has been commonly used to describe the sorption of ions by solids [33]. The general term sorption is used to denote both surface P accumulation on soil components, which may in some cases be accompanied by penetration of the sorbed P by diffusion into the sorbent body [34]. Phosphorus sorption data are usually fitted to the linear form of the Langmuir equation [35]. Although the linear and nonlinear Langmuir equations are mathematically equivalent, there are several limitations to using linearized Langmuir equations; hence, nonlinear model fitting was employed [36]:

$$x = X_{\max}bc/(1 + bc) \quad (1)$$

where  $c$  is the concentration of P in the equilibrium solution ( $\mu\text{g P mL}^{-1}$ ),  $x$  is the amount of P sorbed ( $\mu\text{g P g}^{-1}$  soil),  $X_{\max}$  is the Langmuir sorption maximum ( $\mu\text{g P g}^{-1}$  soil), and  $b$  is a constant related to bonding energy ( $\text{mL } \mu\text{g}^{-1} \text{ P}$ ).

Based on the dual considerations of goodness-of-fit and the statistical behaviour of the Ratkowsky model [37], the Freundlich equation and its modifications are recognised as being superior to other equations for describing phosphate sorption:

$$x = kC^n \quad (2)$$

where  $x$  is the amount of P sorbed ( $\mu\text{g P g}^{-1}$  soil);  $C$  is the equilibrium P concentration in the soil solution ( $\mu\text{g P mL}^{-1}$ );  $k$  is taken as a measure of the abundance of sorption sites (the dimensionless constant); and  $n$  is the slope that is considered to be an indicator of the energy of sorption.

The phosphorus-buffering capacity (PBC) was measured using the Ozanne and Shaw index [38], which is the increase in P sorption for a solution P concentration in-

crement of 0.25 to 0.35  $\mu\text{g P mL}^{-1}$  for an equilibrium solution and may be derived from the Freundlich equation:

$$\text{PBC} = k (0.35^n - 0.25^n) \quad (3)$$

#### 2.4. $^{31}\text{P}$ Nuclear Magnetic Resonance ( $^{31}\text{P}$ -NMR) Spectroscopy

Samples for  $^{31}\text{P}$ -NMR analysis of extractable P were prepared by extracting 5 g of soil with 100 mL of 0.5 M NaOH–0.1 M EDTA solution at room temperature for 16 hrs with occasional stirring. The resultant solution was filtered through a Whatman #41 filter paper. The remainder of the extract was freeze-dried. In total, 1 g of the freeze-dried extract was weighed into a 50 mL plastic centrifuge tube, to which 2.5 mL of  $\text{D}_2\text{O}$  and 0.5 g of solid NaOH were added. The suspensions were vortexed for 2 min, left to stand for 2 h, and centrifuged. The supernatant was transferred into a 10 mm NMR tube and immediately used for NMR spectroscopy [39].

$^{31}\text{P}$ -NMR spectra were obtained at 202.46 MHz with a Bruker ARX-500 high-resolution NMR spectrometer using a pulse angle of  $90^\circ$  with a 0.5 s delay and an acquisition time of 0.655 s. Accumulation time was 16 h. The rate of accumulation was optimised for the extremely low P concentration in some of the samples. Phosphorus compounds were identified by their chemical shift relative to an external orthophosphoric acid (85%) standard. Assignment of peak shifts utilised the criteria of [40,41]. As shown in Figure 1, these are phosphonates (15 to 20 ppm shift), inorganic orthophosphates (5.5 to 7 ppm shift), orthophosphate monoesters (inositol phosphates, sugar phosphates, mononucleotides; 3 to 5.5 ppm shift), orthophosphate diesters (phospholipids, DNA, RNA; 1 to  $-2$  ppm shift), pyrophosphate ( $-3.5$  to  $-5$  ppm shift), and polyphosphates (ATP and ADP;  $-6$  to  $-23$  ppm shift). Peak areas were obtained via instrumental integration. The inorganic orthophosphate and orthophosphate monoester signals were separated using a boundary determined from the valley between the two signals to the baseline [39].

#### 2.5. Physical Properties of Soils

Particle size distribution was determined using the pipette method described in [42]. Ten grams of sieved, air-dried soil was boiled to destroy aggregates and heated with hydrogen peroxide to remove organic matter. A dispersing agent (sodium hexametaphosphate and sodium hydroxide) was added, and the soil was mixed vigorously. The resultant mixture was then placed in a glass column and made up to 500 mL with deionised water. As soil particles settle at different but constant rates depending on their size and ambient temperature, it was possible to collect a sample of the silt and clay fractions, as well as the silt fraction, using a pipette. The mass of the clay and silt fractions were deducted from the total mass to determine the size of the sand fraction (soil moisture and the mass of the dispersing agent were also considered). The soil texture was determined by comparing the per cent of sand, silt, and clay in the material against a standard texture diagram [43]. A single analysis was performed on sample 1 of both soil layers.

Water retention characteristics were assessed on sieved ( $<2$  mm) repackaged samples using a pressure plate apparatus as described in [44]. Sample 1 of each soil layer was used, in triplicate. The materials were packed into retaining rings and wetted up from below for a minimum of 24 h. Water content at saturation (taken to be  $-0.1$  kPa) was determined by drying the soils at  $105^\circ\text{C}$  and calculating the change in mass. The remaining rings were placed inside pressure chambers on porous plates and pressure was applied using compressed air at  $-10$  kPa,  $-100$  kPa,  $-300$  kPa,  $-600$  kPa, and  $-1500$  kPa. The latter ( $-1500$  kPa) is considered to represent a permanent wilting point. In the chamber, the pressure difference across the plate causes water to move from the soil until equilibrium is reached between the soil matrix forces and the applied pressure. The volumetric water content of the soils at various pressures was determined once equilibrium was reached.



### 3. Results

The pH ( $\text{H}_2\text{O}$  and  $\text{CaCl}_2$ ) of the soil layers was moderately acidic and strongly acidic according to the standard classification for the topsoil and the pallid zone, respectively [45]. A revised classification of the soil pH is presented in Table 1, indicating the pH of the soil layers relative to typical jarrah forest soil. In this revised classification, pH 5.5–7.0: normal; pH 5.0–5.5: slightly acidic; pH 4.0–5.0: moderately acidic; and pH < 4.0: strongly acidic. Soil electrical conductivity (EC) was  $49.3$  and  $27.1 \mu\text{S cm}^{-1}$  in the topsoil and the pallid zone, respectively, classified as low according to [46]. The topsoil and the pallid zone had SOC levels of 2.45 and 0.22%, respectively (Table 1).

**Table 1.** Chemical properties and total elemental contents of the topsoil and the pallid zone.

	Topsoil	Pallid Zone
EC ( $\mu\text{S/cm}$ ) <sup>1</sup>	$49.3 \pm 1.0$	$27.1 \pm 0.7$
EC classification <sup>2</sup>	Low	Low
pH ( $\text{H}_2\text{O}$ ) <sup>1</sup>	$5.56 \pm 0.04$	$4.62 \pm 0.02$
pH standard classification <sup>3</sup>	Moderately acidic	Strongly acidic
pH revised classification	Normal	Moderately acidic
pH ( $\text{CaCl}_2$ )	$4.60 \pm 0.02$	$3.92 \pm 0.01$
Soil organic C (%) <sup>4</sup>	2.45	0.22
Total N (%)	$0.08 \pm 0.00$	$0.01 \pm 0.00$
Total Al (%) <sup>5</sup>	3.82	14.5
Total Si (%) <sup>5</sup>	40.5	31.0
Total Ti (%) <sup>5</sup>	0.27	0.31
Total Fe (%) <sup>5</sup>	0.54	0.71
Total Mn ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	31	nd
Total Ca ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	357	71
Total K ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	332	581
Total Mg ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	nd	422
Total P ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	20	96
Total V ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	nd	66
Total Cr ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	nd	49
Total Ni ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	18	38
Total Rb ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	22	29
Total Zr ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	345	261
Total Hf ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	9	9
Total Th ( $\text{mg kg}^{-1}$ ) <sup>5</sup>	21	23
Quartz (%) <sup>6</sup>	98	67
Gibbsite (%) <sup>6</sup>	1	3
Kaolinite (%) <sup>6</sup>	1	30

<sup>1</sup> Measured on a 1:5 soil:water extract. <sup>2</sup> EC classification according to [46]. <sup>3</sup> pH ( $\text{H}_2\text{O}$ ) standard classification based on [45]. <sup>4</sup> Based on the Walkley and Black method [27]. <sup>5</sup> Elemental composition measured by XRF; nd: non-detectible. <sup>6</sup> Material mineralogy detected with XRD.

The results of the XRD (Table 1) indicate that quartz is a large component of both soil layers, particularly in the topsoil with 98%. This is also reflected in the Si content of the soil layers obtained via XRF: 40.5% and 31% in the topsoil and pallid zone layers, respectively. In addition, gibbsite and kaolinite were also identified in very small amounts (except kaolinite in the pallid zone with higher amounts).

Total elemental concentrations of major plant nutrients, as determined via semi-quantitative XRF analysis, are presented in Table 1. Several of the elements were below the detectable limit, shown with “nd” in the table. Calcium was approximately five times higher in the topsoil ( $357 \text{ mg kg}^{-1}$ ) than in the pallid zone ( $71 \text{ mg kg}^{-1}$ ). All other elements were found in higher concentrations in the pallid zone in comparison with the topsoil. In particular, P was five times greater in the pallid zone ( $96 \text{ mg kg}^{-1}$ ) compared with the topsoil ( $20 \text{ mg kg}^{-1}$ ) (Table 1).

$\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  were the most abundantly available cations in both soil layers, while exchangeable K was very low (Table 2). The ECEC of both the topsoil and the pallid zone was low (Table 2). The exchangeable acidity is the sum of the KCl-extractable  $\text{Al}^{3+}$  and  $\text{H}^+$  ions, the major components of soil acidity. The pallid zone had considerably higher exchangeable acidity ( $1.16 \text{ meq } 100 \text{ g}^{-1}$ ) than the topsoil ( $0.39 \text{ meq } 100 \text{ g}^{-1}$ ) (Table 2).

**Table 2.** Range of exchangeable (soluble) elemental concentrations of the topsoil and the pallid zone.

	Topsoil	Pallid Zone
$\text{NO}_3^- \text{-N}$ ( $\text{mg kg}^{-1}$ ) <sup>1</sup>	$6.33 \pm 0.33$	$1.00 \pm 0.00$
$\text{NH}_4^+ \text{-N}$ ( $\text{mg kg}^{-1}$ ) <sup>1</sup>	$9.67 \pm 0.33$	$1.00 \pm 0.00$
Exc. Ca ( $\text{meq } 100 \text{ g}^{-1}$ ) <sup>2</sup>	$2.23 \pm 0.70$	$0.92 \pm 0.12$
Exc. Mg ( $\text{meq } 100 \text{ g}^{-1}$ ) <sup>2</sup>	$0.84 \pm 0.23$	$0.52 \pm 0.03$
Exc. Na ( $\text{meq } 100 \text{ g}^{-1}$ ) <sup>2</sup>	$0.17 \pm 0.05$	$0.09 \pm 0.01$
Exc. K ( $\text{meq } 100 \text{ g}^{-1}$ ) <sup>2</sup>	$0.15 \pm 0.05$	$0.00 \pm 0.00$
Exc. Al ( $\text{meq } 100 \text{ g}^{-1}$ ) <sup>2</sup>	$0.19 \pm 0.04$	$0.78 \pm 0.14$
Al_KCl ( $\text{meq } 100 \text{ g}^{-1}$ ) <sup>3</sup>	$0.32 \pm 0.02$	$1.03 \pm 0.18$
H_KCl ( $\text{meq } 100 \text{ g}^{-1}$ ) <sup>4</sup>	$0.06 \pm 0.02$	$0.13 \pm 0.02$
Exc. Acidity ( $\text{meq } 100 \text{ g}^{-1}$ ) <sup>5</sup>	$0.39 \pm 0.03$	$1.16 \pm 0.19$
ECEC ( $\text{meq } 100 \text{ g}^{-1}$ )	$3.75 \pm 1.03$	$2.67 \pm 0.16$
ESP (%) <sup>6</sup>	4.7	3.9
Extractable P ( $\text{mg kg}^{-1}$ ) <sup>7</sup>	$3.0 \pm 0.0$	$1.0 \pm 0.0$
Extractable K ( $\text{mg kg}^{-1}$ ) <sup>7</sup>	$45.00 \pm 0.015$	$21.33 \pm 3.28$
Extractable S ( $\text{mg kg}^{-1}$ ) <sup>7</sup>	$11.90 \pm 0.50$	$75.33 \pm 2.32$
Extractable Fe ( $\text{mg kg}^{-1}$ ) <sup>7</sup>	$32.72 \pm 1.23$	$10.86 \pm 0.55$
Extractable B ( $\text{mg kg}^{-1}$ ) <sup>7</sup>	$0.25 \pm 0.05$	$0.25 \pm 0.05$
Extractable Cu ( $\text{mg kg}^{-1}$ ) <sup>7</sup>	$0.16 \pm 0.01$	$0.02 \pm 0.00$
Extractable Mn ( $\text{mg kg}^{-1}$ ) <sup>7</sup>	$4.79 \pm 0.25$	$0.04 \pm 0.00$
Extractable Zn ( $\text{mg kg}^{-1}$ ) <sup>7</sup>	$0.23 \pm 0.09$	$0.02 \pm 0.01$

<sup>1</sup> 2 M KCl-extractable, colourimetrically determined using the method of Keeney and Nelson [28]. <sup>2</sup> Based on the Gilman and Sumpter method [26]. <sup>3</sup> KCl-extractable  $\text{Al}^{3+}$ . <sup>4</sup> KCl-extractable of  $\text{H}^+$ . <sup>5</sup> Exchangeable acidity =  $\text{Al\_KCl} + \text{H\_KCl}$ . <sup>6</sup> Exchangeable sodium percentage (ESP) =  $\frac{\text{Exc.Na}}{\text{ECEC}} \times 100$ . ESP > 6% indicates a sodic soil according to Australian soil classification [43]. <sup>7</sup> Extractable nutrient concentrations are an indication of the plant-available pool.

Total N concentrations of <0.15% are classified as low [47], and both soil layers had concentrations well below this critical value (Table 1). Soil  $\text{NO}_3^- \text{-N}$  and  $\text{NH}_4^+ \text{-N}$  were  $6.33$  and  $9.67 \text{ mg kg}^{-1}$  in the topsoil, respectively, while they were much lower in the pallid zone ( $1 \text{ mg kg}^{-1}$ ) (Table 2). Both soil layers had also very low levels of extractable P (< $3 \text{ mg kg}^{-1}$ , Table 2) (classified according to [47]). Mean copper concentrations were  $0.16$  and  $0.02 \text{ mg kg}^{-1}$  in the topsoil and the pallid zone, respectively, considered low. The manganese concentration was considerably higher in the topsoil ( $4.79 \text{ mg kg}^{-1}$ ) than in the pallid zone ( $0.04 \text{ mg kg}^{-1}$ ) (Table 2). Sulphur (S) levels of both soil layers were higher than the critical value of  $5\text{--}10 \text{ mg kg}^{-1}$ , being  $11.9$  and  $75.33 \text{ mg kg}^{-1}$  in the topsoil and the pallid zone, respectively (Table 2).

The Langmuir P maximum ( $X_m$ ) values were  $67.55$  and  $40.21 \mu\text{g P g}^{-1}$  in the topsoil and the pallid zone, respectively, classifying them in the low-to-moderate P sorption soils of Southwestern Australia. The P adsorption lines for the two soils (Figure 2) illustrate that the soils are slightly different in P-buffering capacity, with topsoil showing higher P-buffering capacity compared with the pallid zone (Table 3).

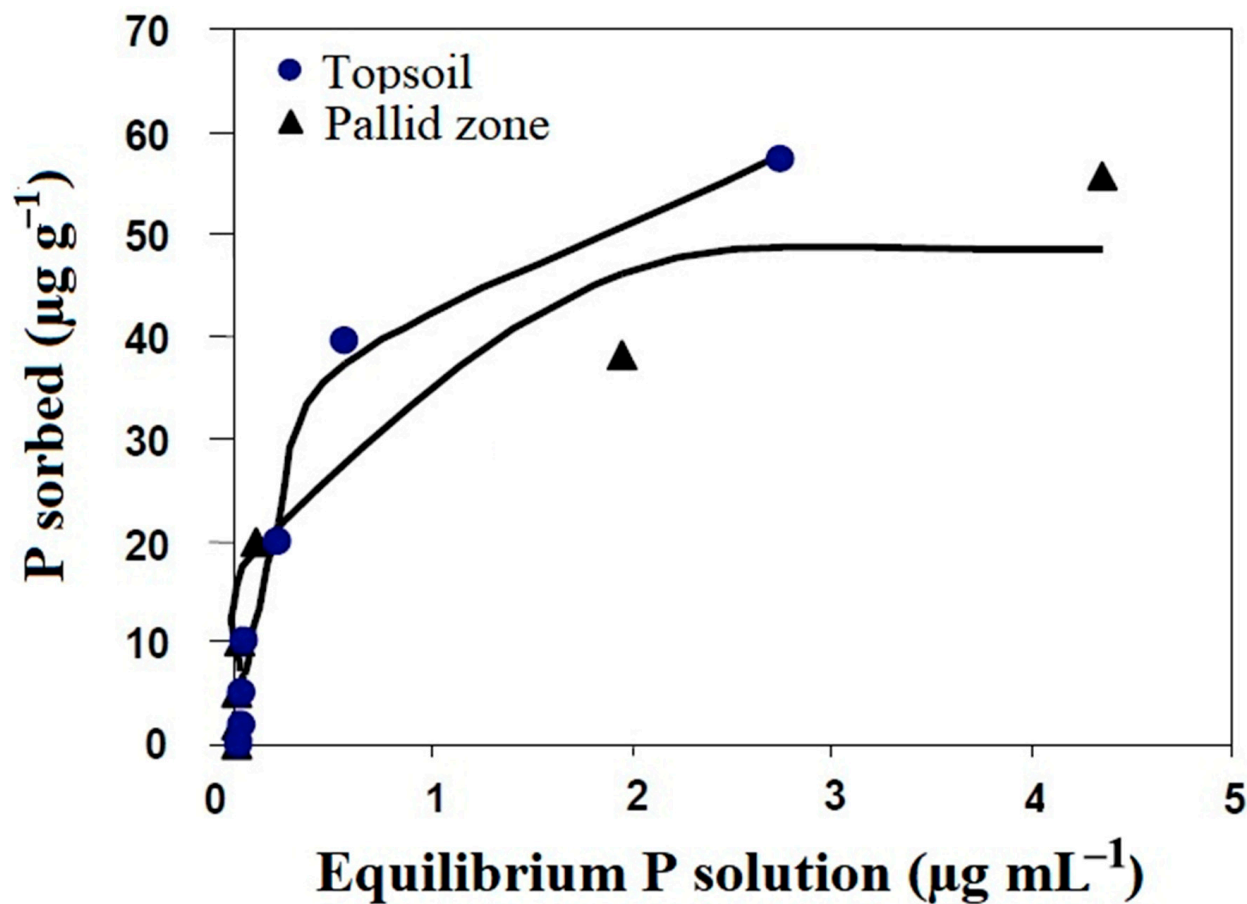


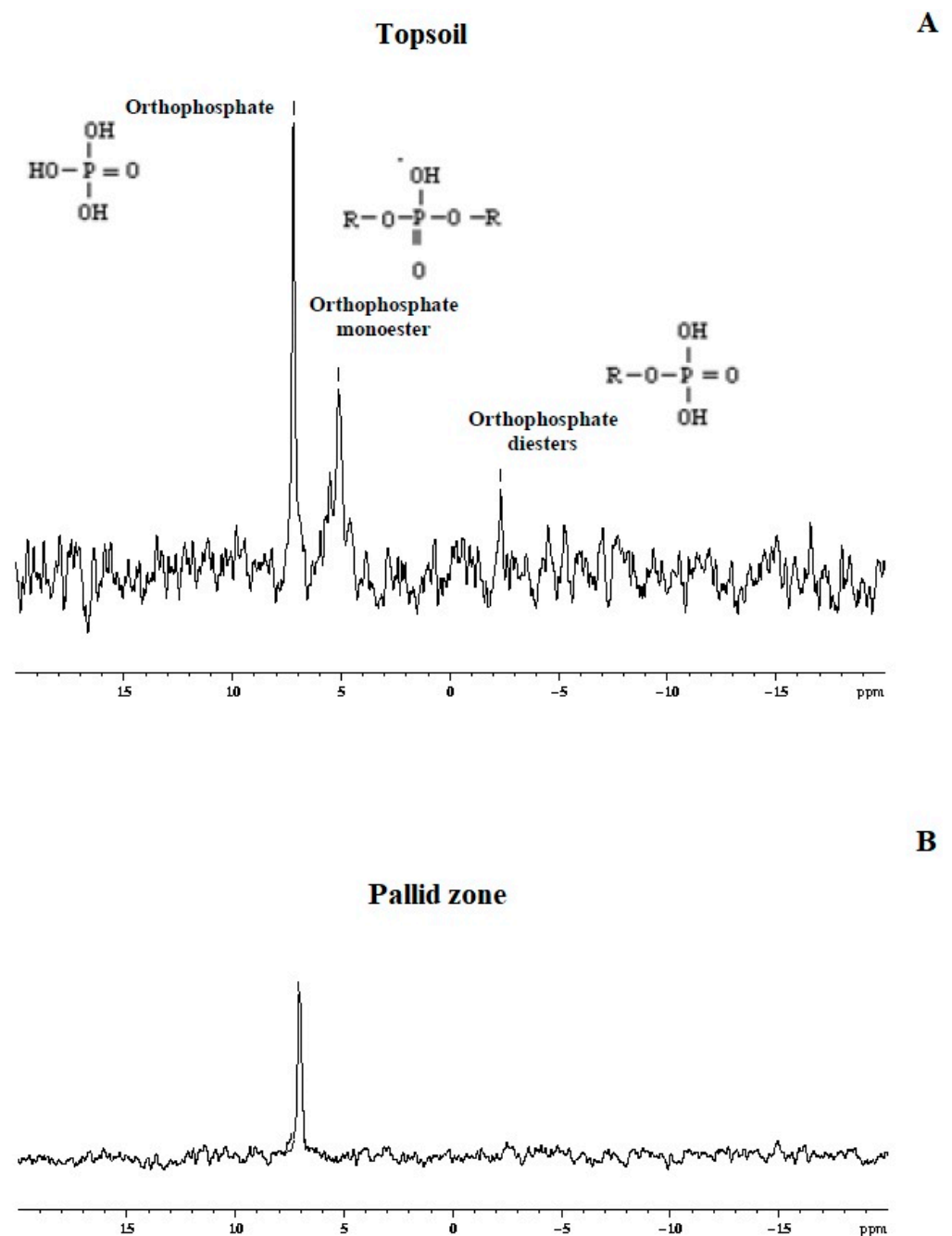
Figure 2. Phosphorus adsorption in two soil layers (topsoil and pallid zone).

Table 3. Langmuir and Freundlich coefficients and phosphorus-buffering capacity (PBC) using the Ozanne and Shaw (1967) index for the topsoil and the pallid zone.

Parameters	Pallid Zone				Topsoil			
	Langmuir Parameters		Freundlich Parameters		Langmuir Parameters		Freundlich Parameters	
	<i>b</i> (mL µg <sup>−1</sup> )	<i>X<sub>m</sub></i> (µg g <sup>−1</sup> )	<i>k</i> (µg g <sup>−1</sup> )	<i>n</i> (mL µg <sup>−1</sup> )	<i>b</i> (mL µg <sup>−1</sup> )	<i>X<sub>m</sub></i> (µg g <sup>−1</sup> )	<i>k</i> (µg g <sup>−1</sup> )	<i>n</i> (mL µg <sup>−1</sup> )
Initial estimates	7.12	40.83	35.46	0.38	1.50	72.20	41.91	0.38
Fitted values	8.72	40.21	30.98	0.34	2.18	67.55	38.44	0.45
Standard errors	1.51	2.08	3.01	0.06	0.39	4.27	3.64	0.08
Approx 95% confidence limits								
Lower bound	3.92	33.60	21.40	0.14	1.09	55.78	28.33	0.23
Upper bound	13.52	46.82	40.57	0.55	3.27	79.33	48.55	0.66
P-buffering capacity (µg g <sup>−1</sup> )			2.34				3.37	
Goodness of fit statistics								
Sum of standard errors (SEE)		130.52		59.12		28.32		171.59
Model efficiency		0.94		0.97		0.99		0.93
Akaike's information criterion (AIC)		36.48		31.73		27.31		38.12

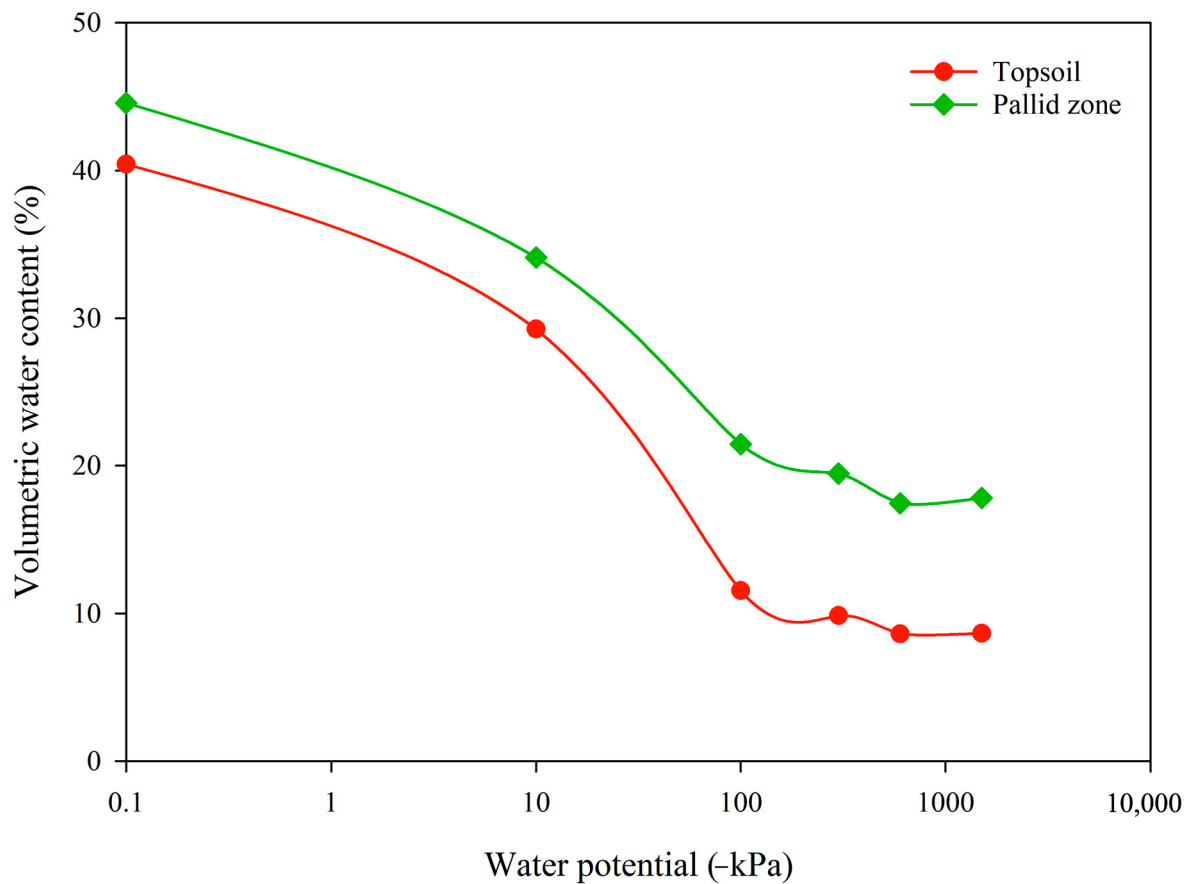
A solution <sup>31</sup>P-NMR analysis of 0.5 M NaOH–0.1 M EDTA extracts identified structural forms of inorganic and organic P. The abundance of all the P groups was determined using <sup>31</sup>P-NMR. Orthophosphate was higher for the topsoil compared with the pallid zone. Monoester P and diester P were also detected only in the topsoil (Figure 3).





**Figure 3.** Solution NMR spectra obtained for NaOH/EDTA extracts of the topsoil (A) and pallid zone (B).

The <2 mm fraction constituted the majority of both soil layers by mass (Table 4). The fine earth of the topsoil and the pallid zone was classed as sandy loam and clay loam, respectively (Table 4). Topsoil had a large sand component (>86%), suggesting it should be well drained. The topsoil was classed as having moderately rapid drainage based on laboratory testing (Table 4). Using the second constant head technique, hydraulic conductivity in the pallid zone ( $266 \text{ mm day}^{-1}$ ) was ~10 times lower than the topsoil ( $2539 \text{ mm day}^{-1}$ ) (Table 4). The soil water retention curves (Figure 4) are consistent with the soil textures (Table 4), meaning that the pallid zone with finer texture had a higher water retention capacity. Soil bulk density was  $1.9$  and  $1.4 \text{ g cm}^{-3}$  in the topsoil and the pallid zone, respectively (Table 4).



**Figure 4.** Soil water retention curve in two soil layers (topsoil and pallid zone); FC is field capacity and PWP is permanent wilting point.

**Table 4.** Physical properties of the topsoil and the pallid zone.

	Topsoil	Pallid Zone
Material < 2 mm (%)	64.8 ± 12.8	80.6 ± 2.6
Clay (%)	9.6	25.8
Silt (%)	4.2	23.1
Sand (%)	86.2	51.1
Soil texture <sup>1</sup>	Sandy loam	Clay loam
Average k-sat (mm day <sup>-1</sup> ) <sup>2</sup>	2539 ± 686	266 ± 114
Drainage class <sup>2</sup>	Moderately rapid	Moderate
Bulk density (g cm <sup>-3</sup> )	1.9	1.4

<sup>1</sup> Soil textural classes based on [43]. <sup>2</sup> Saturated hydraulic conductivity (k-sat) of materials as determined using the alternative constant head technique and the associated drainage class.

## 4. Discussion

### 4.1. Basic Properties of Soils

A brief review of the available data suggests that the soils of the jarrah forest are typically slightly acidic, often in a range of pH 5.5–6.5 [48–50]. Based on the revised classification system, indicating the pH of the tested soil relative to typical jarrah forest soil, the topsoil and the pallid zone were found to have normal and moderately acidic pH levels, respectively. The pH of both soil layers did not appear to limit the growth of native vegetation, except acid-sensitive species. The salinity of both soil layers was low; therefore, salinity is not expected to be a concern for plant growth. The exchangeable sodium percentage also indicated that none of the soil layers were sodic (threshold ESP 6% [51]). In 1989, it was estimated that about 2% of agricultural land in the jarrah forest region of

Australia was too saline for crop production [52]. Forest over-clearing in Southwestern Australia resulted in biodiversity loss and major hydrological changes, which consequently increased soil salinity in some agricultural areas [53], but this was not an issue of concern in our soils.

Soil organic carbon is determined via land management, soil type, climate, mineralogy, vegetation, and topography [54]. Organic carbon is typically the greatest at the soil surface, decreasing exponentially with depth [54]. In agricultural systems, SOC levels above 2% in the A horizon are considered high [55]. However, agricultural systems are cultivated and highly disturbed and cannot be compared to natural forest systems. Ref. [56] measured the same amount of soil organic carbon in the same jarrah forest in southwest Western Australia as we recorded in our study area. They also observed decreased soil organic carbon content with depth, reaching 0.58% at a depth of 1 m. [57] observed soil carbon development with an increasing restoration age in Southwestern Australia, and they also recorded decreased C content with increasing soil depth.

The mineralogy of both soil types was dominated by quartz. The abundance of quartz in both soil layers has led to high Si content. Kaolin minerals, which are aluminosilicates, often make up the majority of clays in the pallid zone layer [58] and are formed from the weathering of acid rocks in non-alkaline conditions. The presence of high concentrations of kaolinite in the pallid zone indicates strong weathering and leaching [55]. Higher Al content in the pallid zone layer compared with the topsoil can be related to much higher kaolinite in this layer. Gibbsite, which is found in association with kaolinite, is a common aluminium oxide [59]. Gibbsite is commonly found in small-to-trace amounts in Western Australian soils but can be the predominant mineral in bauxite deposits [55]. High kaolinite content in the pallid zone layer can retain large amounts of nitrate and make N inaccessible to most field crops since it is deposited in deep layers [60]. Furthermore, Al originating from kaolinite fixes phosphates with Al-OH groups, generally making P unavailable for plant uptake [61] unless roots have special adaptations, as found in some jarrah forest species, particularly in Proteaceae and Myrtaceae [62].

#### 4.2. Chemical Fertility

The cation exchange capacity is a measure of the total capacity of soil to hold exchangeable cations and, therefore, indicates the negative charge present per unit of mass of the soil [63]. It is also an indirect measure of soil fertility, as it provides an indication of the ability of soils to retain ions important in plant nutrition [64]. The effective cation exchange capacity (ECEC), which is the sum of all exchangeable cations, is largely related to clay minerals, soil pH, and the amount of organic matter contained in the soil [65]. As a general rule, soils with little organic matter and low clay content have a lower ability to hold exchangeable cations than heavier, fine-textured soils rich in organic matter. Therefore, coarse materials such as silica sands usually have a low cation exchange capacity, while minerals such as illite have higher capacities [26]. However, the type of clay mineral present in the soil is also important. Kaolin and gibbsite have negligible cation exchanges from permanent charge surfaces because of isomorphous substitution [26], and the lower ECEC in the pallid zone layer may be related to its higher kaolinite content (30%) compared with that in the topsoil (1%). Low soil pH is also associated with a lower cation exchange capacity, as fewer cations are available in increasingly acidic conditions. The ECEC of both soil layers was low, suggesting they are relatively infertile.

Nitrogen is an important plant nutrient, and the mineral N component of the soil is primarily  $\text{NH}_4^+$  or  $\text{NO}_3^-$ . The total N provides an indication of the N that may be mineralised under appropriate conditions, and values for Australian soils can range from <0.02% in subsoils to >2.5% in peats [26]. A great amount of N is held in soil organic matter in Southern Australian forests [66]. A soil nitrate concentration of  $45 \text{ mg kg}^{-1}$  is a reported critical value, resulting in a 90% relative yield of wheat in the >450 mm rainfall zone [67]. Both the topsoil and the pallid zone layers had nitrate levels well below this, but it is difficult to predict whether this will be limiting in a developing native vegetation community that

would typically be adapted to lower levels of N. Ref. [20] observed that N is a limiting nutrient in these soils when P is applied, shifting a P-limited to a N-limited ecosystem. It is worth mentioning that N fixation by leguminous species provides a significant amount of N for plant growth in these ecosystems, reaching  $14 \text{ kg N ha}^{-1} \text{ yr}^{-1}$  [68,69]. In N-poor ecosystems, some native plants may utilise organic N besides  $\text{NH}_4^+$  and  $\text{NO}_3^-$  [70].

Extractable nutrient concentrations are an indication of the plant-available pool and can highlight potential deficiencies or toxicities, but they do not provide definitive levels of plant-available nutrients. Published critical values for soil nutrient concentrations pertain almost exclusively to cultivated crops and have limited relevance to Australian native plants. It is, therefore, difficult to determine whether the nutrient levels presented here are likely to limit the growth of native plant species. The levels of extractable K in both the topsoil and the pallid zone layers were low ( $<70 \text{ mg kg}^{-1}$ ) according to [47]. Boron (B) is an important micronutrient and critical values for deficiency range from 0.15 to  $0.5 \text{ mg kg}^{-1}$ , while values of  $>3 \text{ mg kg}^{-1}$  are indicative of areas at risk of B toxicity [71]. Based on this, the B levels in the two soil layers may be borderline deficient for some plant species. Copper (Cu) is an essential component of many plant enzymes and is involved in providing structural strength through lignification [72]. Copper deficiencies are widespread in native Western Australian soils, and published critical values for crop production vary widely and are dependent on the plant species, the soil type, and the extraction method used. Copper levels  $>0.3 \text{ mg kg}^{-1}$ , determined through extraction with a chelating agent (EDTA), have been found to be adequate for the grain yield of wheat [73], but no information is available on critical Cu levels for jarrah forest species. Both soil layers in our study are Cu-deficient for field crops. Manganese (Mn) levels were also determined, but it is not possible to predict, based on soil testing alone, whether Mn deficiency or toxicity can be expected in the field. Soil Mn concentrations can be greatly affected by site history, field conditions at the time of sampling, environmental conditions such as soil moisture and aeration, the date of sampling, drying conditions, and soil storage, all of which impact the degree of reduction in the soil and, hence, plant availability [74]. Plant-available zinc (Zn) concentrations were also estimated using DTPA extraction. This is the most commonly used method for determining soil Zn levels, but there is little information available on critical levels. Furthermore, different plants vary widely in their abilities to tolerate Zn deficiency and toxicity [75]. The levels of Zn in both soil layers were almost all below an uncalibrated critical level for crop production ( $0.3\text{--}0.8 \text{ mg kg}^{-1}$ ), as suggested by [76], but it is unclear whether these levels will be deficient for Australian native plants. Combined deficiency in Zn and Cu was previously reported by [77] for older and sandier soils such as in the Esperance sandplain of Western Australia, which we may also observe in the Darling Plateau.

#### 4.3. Phosphorus Fertility

Phosphorous is a critical plant nutrient and is limited in most Western Australian soils. Australian soils typically contain low P ( $<0.2\%$ ) unless fertilised, and then, much of this is fixed by Fe and Al (hydr)oxides. In these ecosystems, diverse native plant communities adapted to survive in very low-P soils can be found [56]. Higher contents of total P in the pallid zone compared with the topsoil may be related to the higher clay content of this soil and the long-term leaching of P. It should also be appreciated that the XRF results indicated total, not available, concentrations. An understanding of P sorption and release characteristics in soils is important when interpreting soil P availability to plants. Langmuir P maximum ( $X_m$ ) values ranged between 11 and  $2132 \text{ } \mu\text{g P g}^{-1}$ , with a median value of  $200 \text{ } \mu\text{g P g}^{-1}$  for diverse Western Australian virgin soils [78]. The large variation in the P-buffering capacity (PBC) of the two soil layers is probably a reflection of the variation in soil properties, and much of this difference may be due to the organic matter content of the soils. PBC usually has a positive linear relationship with clay, silt, pH (NaF), organic C, and the various forms of extractable Fe and Al. The PBC of a given soil has large effects on both the amount of phosphate fertilizer needed to produce maximum plant yield and

on the critical values used to interpret soil tests. From a fertility point of view, based on the slightly higher P-buffering capacity for the topsoil layer, it has a slightly higher potential for adsorbing and releasing anions such as phosphate ions (reversible available P) compared with the pallid zone layer. According to [79], the amount of available P can be between 1 to 10% of the total P in Australian soils. In the topsoil layer, 15% of the total P is plant-available, while in the pallid zone layer, only 1% of the total P is available P.

Monoester phosphates tend to accumulate in soils because of the slow rate of decomposition compared with many other esters and partly because of their strong sorption onto mineral components. The orthophosphate monoester functional group, which includes inositol phosphates, sugar phosphates, and mononucleotides, generally constitutes up to 60% of total organic P in soils. Diester phosphate is the fraction that is subjected to rapid mineralization in comparison with monoester P, and this is considered to be responsible for most of the rapid increase in the labile inorganic P fraction [80,81]. Some organic P, especially the diester form, may also be hydrolysed during NaOH extraction. The absence of organic P content in the pallid zone layer is most probably because of a lack of appreciable organic matter. This will mean the supply of mineralizable P must come only from the topsoil layer. The huge difference in the supply of  $\text{PO}_4^-$  ions (orthophosphate peak) supports this interpretation. This shows the importance of the topsoil layer in plant nutrition in these soils when inorganic fertilisers are not applied.

#### 4.4. Key Physical Properties

The topsoil has a sandy loam texture. It is expected to drain well because of the large amount of quartz sand and be structurally stable with no tendency to hardset. The pallid zone in the clay loam textural class is moderately well drained [82] and susceptible to mechanical compaction, but the clay content is not too high to restrict plant root growth [43], especially of native species [83]. This layer is rich in kaolinite, which tends to decrease aggregate stability. Based on the low saturated hydraulic conductivity ( $266 \pm 114 \text{ mm day}^{-1}$ ) of the pallid zone layer, this poorly structured layer drains slowly and, hence, the topsoil is rapidly saturated under average rainfall conditions, presenting problems with water-logging and runoff that are the most likely impediment to plant growth.

The matric potential reflects the particle size distribution of the samples, with the finer-textured soils holding a greater volume of water at each measurement potential. The difference between the  $-1500 \text{ kPa}$  and the  $-10 \text{ kPa}$  water contents is generally recognised as the amount of plant-available water; however, this is based on evidence from agricultural systems. Since soil matric potential indicates how much energy plants will have to exert to extract water molecules from soil particles, it can be different between native and crop plants. Native plant species are typically able to extract water from soils with much greater matric potentials [84]. The topsoil seems to show a good capacity to hold and release water to plants over a wider range of matric potential, enhancing its suitability as a plant growth substrate in restoration schemes. This is important since even native drought-tolerant plant species should be provided with enough water at the early stages of growth in restoration sites. It should be noted that soil water retention curves will vary with bulk density. Soils with higher bulk density cannot hold a large amount of water and have a lower amount of total available water, which is the difference between soil water content at field capacity and the permanent wilting point.

## 5. Conclusions

The two jarrah forest soil layers considered in the current study were highly contrasting in many key prospective properties for use in land rehabilitation. Each offered its own unique characteristics, outlined here in detail for the first time. We found the capacity of these soils to provide a stable growth medium differed considerably regarding their ability to retain and supply nutrients. The pH of both soil layers is not a limiting factor for the growth of native vegetation, except in the pallid zone for acid-sensitive species. The salinity of both soil layers was also low, and it is not a concern for plant growth. Quartz is the



dominant mineral in both soil layers, leading to high silicon content. High kaolinite content in the pallid zone layer can retain large amounts of nitrate and phosphorus, which makes nitrogen inaccessible to most field crops and phosphorus unavailable for both native and field crop species. Nitrogen concentrations in both soil layers show that they are nitrogen-deficient for field crops, but it is difficult to predict whether this will limit a developing native vegetation community that is generally considered P-limited [85]. Although the pallid zone has higher total phosphorus compared with the topsoil, the topsoil has a higher potential for adsorbing and releasing phosphate ions because of its higher phosphorus-buffering capacity. The  $^{31}\text{P}$ -NMR results also showed the importance of organic P in the topsoil for ecosystem nutrition when inorganic fertilisers are not applied in state-of-the-art restoration schemes. The low effective cation exchange capacity of both soil layers indicates that they are infertile. Considering micronutrients, boron, copper, and zinc in the two soil layers may be deficient for some plant species. The pallid zone layer drains slowly, indicated by its low saturated hydraulic conductivity, leading to the rapid saturation of the topsoil and water-logging and runoff under average rainfall conditions, which are the most likely impediment to plant growth. Overall, we found that the topsoil material was ideally suited as a plant growth medium for native plants in low- or no-input systems and that pallid zone saprolite clays have good physical properties for geotechnical uses in landscape design.

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**Data Availability Statement:** The data presented in this study are available in this article.

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